Electrochemical studies of arabic gum as a corrosion inhibitor for metallic structure steel in a hydrochloric acid solution

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Abstract - The inhibition effect of arabic gum, a natural product Acacia Senegal, on the corrosion of metallic structural steel S235 in the hydrochloric acid medium was studied using potentiodynamic polarization, spectroscopy measurements electrochemical impedance.
Arabic gum gave an inhibition efficiency of 83% for a mass concentration of 0.3% at the ambient temperature. The results obtained from the various corrosion assessments are a good combination. Polarization measurements indicate that arabic gum is a mixed type inhibitor. The adsorption of natural compound on the surface of the steel, in a hydrochloric acid solution, obeys the Langmuir adsorption isotherm.

Keyword - adsorption, hydrochloric acid, S235 steel, corrosion, arabic gum.

1. INTRODUCTION

The structure or morphology of units designed based on metallic materials such as cast iron, steel, etc. can be damaged when exposed in specific environments. This harmful phenomenon takes the name of corrosion and corresponds to the deterioration of a metal or alloy by an attack or a chemical reaction with a corrosive environment. It is a recurring problem and difficult to eliminate completely, thermodynamic metals tend to return to their mineral forms (oxide, sulfide, carbonate, nitrate, etc.) much more stable.
Due to the general aggressiveness of acid solutions, the use of inhibitors to control the destructive attack of acidic environments has been found to have widespread applications in many industries [1]. Recently, the inhibition of corrosion of steel in acid solutions by different types of organic inhibitors has been widely studied [2] - [6]. Organic nitrogen-containing compounds are known to be effective corrosion inhibitors in hydrochloric solutions, while sulfur-containing compounds are sometimes preferred for sulfuric acid solutions [7].

The choice of arabic gum, a natural product Acacia enegal, very abundant in the southern and eastern area of Senegal, as a corrosion inhibitor is based on high solubility in acidic environments. The action of this inhibitor depends on the interaction between the functional groups and the metal surface.

Works have been carried out on N-heterocyclic inhibitors on mild steel in an acid environment [8] - [13]. Likewise, in the literature [14] - [18], we find compounds such as 5-acetyl-6-methyl-4- (4-nitro phenyl) - 3, 4-dihydropyrimidin-2 (1H) -one (DHPM-1), 5-acetyl-6-methyl-4- (3-nitro phenyl) -3, 4-dihydropyrimidin-2 (1H) -one (DHPM-2), and 5-acetyl-6-methyl- 4-phenyl-3, 4-dihydropyrimidin-2 (1H) -one (DH PM-3) were used as corrosion inhibitors.

This work studies the inhibition of corrosion of S235 steel in hydrochloric acid (HCl) of 1.0 M molar concentration by arabic gum. The study was carried out using polarization measurements, electrochemical impedance spectroscopy.

2. EXPERIMENTAL METHODS

The electrochemical experiments were carried out using a cell with three electrodes at the ambient temperature. Steel S235, of percentage by mass of elements other than iron C = 0.17, Mn = 1.40, N = 0.012, P = 0.04, S = 0.04, Cu = 0.55, is a material for construction of the conventional elastic limit minimum 235 N / mm^2 or (Mpa). The samples were cut in the form of plates of exposed surface 3.6 cm^2, coated in resins of the "Epoxy Steel" type, were used for the electrochemical measurements. A platinum grid as a counter electrode, an Ag / AgCl reference electrode and the working electrode made up of the sample.
The sample undergoes a pretreatment, which consists of polishing the surface of the metal with sandpaper of

increasingly fine particle size (180-320-400-600-1200), washed with distilled water and rinsed with l acetone then dried in the open air. All the electrochemical measurements were carried out using a palmsens 4 brand potentiostat controlled by the PSTrace 4.8 software.

The electrolyte used is a hydrochloric acid (HCl) solution with a concentration of 1 M. This electrolyte is obtained by diluting in distilled water a commercial hydrochloric acid solution whose mass percentage is 37% and density 1.18.

For each test, an amount of arabic gum ranging from 0% to 0.6% is added relative to the volume of acid used to monitor the behavior of the metal and the inhibitory effectiveness.

The inhibitor used for this study is arabic gum. The natural product, Acacia Senegal, very abundant in the south and east of Senegal. Arabic gum is a complex polysaccharide which is in the form of a branched-chain. It is a compound; either neutral or slightly acidic, which is in the form of a mixed salt (calcium, magnesium and potassium) of polysaccharide acid. Fig. 1 presents the molecular structure of gum arabic [19].
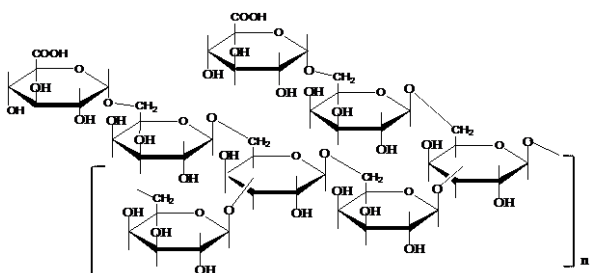

Fig.1. Chemical structure of the arabic gum

The polarization curves of Tafel were obtained by the potentiodynamic method, the scanning speed is fixed at 0.5 mV / s and the equilibrium time at 30 minutes. The working electrode is kept immersed beforehand at the free corrosion potential for 1 hour under magnetic stirring.

Electrochemical impedance spectroscopy (EIS) measurements were performed under potentiostatic conditions in a frequency range from 5000 Hz to 100 mHz, with a signal amplitude of 10 mV. All the experiments were measured after immersion of the steel for 3 h of immersion at ambient temperature at the abandonment potential in the frequency range, in 1.0 M HCl in the absence and in the presence of various concentrations of inhibitors.

3. RESULTS AND DISCUSSION
3.1. Polarization measurements
Fig. (2) shows the effect of the concentration of arabic gum on the anodic and cathodic potentiodynamic polarization characteristics of S235 steel in a 1 M HCl solution at a scanning speed of 0.5 mV / s and the equilibrium time 30 minutes and at the ambient

temperature. The results clearly show that the addition of arabic gum slightly displaces the corrosion potential (E_{corr}) in the cathodic direction and reduces the anodic and cathodic current densities. However, the cathode current is reduced more significantly than the anode current.
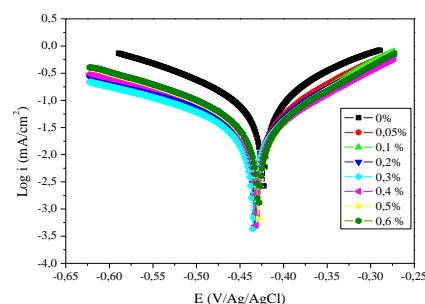

Fig. 2. Stationary polarization curves of S235 steel in HCl (1M), at different concentrations of arabic gum inhibitor

It is observed that there is a small variation in the corrosion potential as a function of the concentration of the inhibitor. The inhibitory nature of arabic gum is therefore mixed. Indeed, it is established that an inhibitor can be classified as cathodic or anodic type if the displacement in E_{corr} during the addition of the inhibitor is more considerable than 85mV compared to the corrosion potential of steel in uninhibited medium [20], [21]. On the other hand for our study, the maximum displacement is 9 mV.

The electrochemical parameters (i_{corr}, E_{corr}, b_c, b_a and R_p) associated with polarization measurements of arabic gum at different concentrations were simultaneously determined and listed in the table (1). The experimental results also show that there is a slight displacement of b_c and b_a in the HCl solution (1M). It can therefore be suggested that the corrosion mechanism has not changed with the addition of the inhibitor [22].

We also note that the cathode Tafel lines are parallel, indicating that the process follows a charge transfer mechanism [23]. According to the data in the table (1), it is evident that the slopes of the anode (b_a) and cathode (b_c) lines of Tafel remain almost constant when the gum is added. These results indicate that this inhibitor works by merely blocking the surface available for the anodic and cathodic processes. In other words, the inhibitor decreases the corrosion surface without affecting the corrosion mechanism and does not cause the inactivation of part of the surface relative to the corrosive medium.

The introduction of the inhibitor into the acid solution hydrochloric acid does not modify the evolution and reduction mechanism of hydrogen on the surface of the steel. The sharp decrease in current density respectively without the inhibitor (687,421 μA / cm^2)

and with 0.3% of this product (112,620 μA / cm^2) is due to an inhibitory

activity of arabic gum on the steel interface.

C (%)	E$_{corr}$ (mV)	ΔE (mV)	i$_{corr}$ (μA/cm^2)	R$_p$ (Ω)	b$_a$ (mV/dec)	b$_c$ (mV/dec)	V$_{corr}$ (mm/year)	IE (%)
0	-0.426	0	687.421	27.407	70	114	4.720	-
0.05	-0.429	3	187.023	96.624	64	119	1.287	72.79
0.1	-0.432	6	156.012	114.378	76	110	1.024	77.25
0.2	-0.433	7	124.633	129.704	60	98	0.807	81.86
0.3	-0.435	9	112.620	132.582	52	102	0.778	83.61
0.4	-0.432	6	141.331	110.652	64	108	0.936	79.44
0.5	-0.431	5	176.168	100.820	61	124	1.163	74.37
0.6	-0.431	5	226.697	80.071	65	117	1.474	67.02

Table (1) The electrochemical parameters associated with polarization measurements for S235 steel in a 1M HCl solution in the absence and presence of different concentrations of arabic gum.

The inhibition efficiencies at different concentrations of arabic gum are calculated and grouped in the table (1), using the equation below [24]:

$$IE = \left((i^0_{corr-i_{corr}})/i^0_{corr} \right) \times 100 \qquad (1)$$

i^0_{corr} and i_{corr} being the corrosion current densities respectively with and without inhibitor

The inhibitory power increases with the concentration of the substance and reaches a maximum value of 83.24% for a content of 0.3% in arabic gum. This inhibition rate is very satisfactory and modifies the electrochemical behaviour of steel in the vicinity of Ecorr. All of the electrochemical parameters drawn from these curves are grouped in the table (1).

The inhibitors have an action on the reduction of the current on the anodic part of the surface of the metal. If this blockage is only partial, it can locally cause an increase in the current density on these surfaces. It can lead to a localized corrosion process, more intense than in the absence of an inhibitor, hence the importance of the active element content. Inhibitors act by forming a passive layer and must be used with caution because they can cause widespread initial corrosion [25], [26]. In the field of low anode potentials, the density of the anode current increases slightly with the overvoltage. When the desorption potential (E$_d$) (or non-polarizability) [27] - [29] is exceeded, arabic gum has almost no effect on the anode curves; the anode current intensity then increases rapidly and the metal dissolves with a steeper slope in the region of high potentials. The sharp increase in anodic current density after the desorption potential is attributed to the desorption of the arabic gum molecules adsorbed on the surface of the steel. This clearly shows that the mode of inhibition of this compound depends on the electrode potential. The mode of protection observed is then described as resulting from the formation of a layer of the inhibitor adsorbed on the surface of the electrode [30].

Note also that the values of the desorption potential are almost constant (around –300 mV / Ag / AgCl) for the different gum contents studied. This leads us to believe that the values of the desorption potential of the gum are slightly modified by the overlapping of the molecules of the inhibitor. This result is in agreement with the literature [31].

However, beyond the optimal inhibitor content, an increase in the corrosion rate is observed. This phenomenon can be due to the formation of deposits of insoluble corrosion products, the local conditions being considerably modified and the damage mechanisms can evolve and therefore the phenomenon of corrosion continues [32]. This continued corrosion beyond the optimal concentration of arabic gum leads to a new shift in potential towards anode values.

The anodic polarization curves show a decrease in current densities. However, whatever the proportions of the eraser, the current densities are quite comparable.

The modification of the cathode Tafel slopes, in the absence and the presence of the inhibitor, shows that the reduction reaction on the surface of the steel is not modified by the addition of gum and that it is carried out according to a genuine activation mechanism. The inhibitor is first adsorbed on the surface of the steel before acting by merely blocking its active sites. Cathodic inhibitors induce an increase in cathodic overvoltage, and therefore reduce the corrosion current. If this inhibitor does not entirely stop the corrosion reaction, it does not however present the danger of localized corrosion.

Arabic gum is a highly water-soluble polysaccharide containing a hydroxyl functional group (-OH) and a

carboxyl functional group (-COOH) [33]. In acid solution such as 1M hydrochloric acid (HCl) solution, the carbonyl compound (C = O) can be protonated and the molecule then exists in the form of polycation. The anions of Cl^- tend to be adsorbed on the surface of the steel, which leads to a negative charge on the surface

3.2. Electrochemical impedance spectroscopy
3.2.1. Effect of the concentration of arabic gum
In order to study the adsorption and inhibition mechanism of arabic gum, electrochemical impedance spectroscopy measurements were carried out to study the acid solution characteristic of the steel / electrolyte solution interface of S235. Fig. 3 shows the impedance spectra of the S235 steel obtained at the open circuit potential after an exposure period of 3 h in a 1 M solution of hydrochloric acid (HCl) in the absence and in the presence of different arabic gum concentrations. Analysis of the data reveals that the impedance diagram obtained with 1 M HCl shows only one high-frequency capacitive loop. The same trend of the single capacitive loop was also observed for S235 steel immersed in 1 M HCl containing different concentrations of arabic gum. The shape of the capacitive loops suggests that the

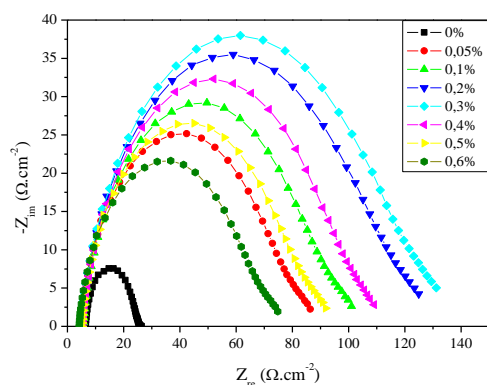charge transfer controls the corrosion of S235 steel at the corrosion potential (E_{corr}) [34].

Fig. 3. Nyquist diagrams of corrosion of S235 steel in 1M HCl acid at different concentrations of arabic gum

On the other hand, the diameter of the semicircle gradually increases with the increase in the concentration of arabic gum up to the value 0.3% and

of the steel. The formation of positively charged protonated species facilitates the adsorption of the compound on the surface of the metal by the electrostatic interaction between the arabic gum molecule and the surface of the steel by physisorption.

consequently the inhibition efficiency increases. However, beyond this concentration value (0.3%), the diameter of the half-loop decreases as does the inhibitory power. Regarding the inhibition process, it is generally accepted that adsorption of inhibitors at the metal / aggressive solution interface is the first step in the inhibition mechanism [35]. Given the dependence of the inhibition efficiency on the inhibitor concentration as shown in fig. 3, it seems possible that this inhibitor acts by adsorbing and blocking the active centre available on the surface of the steel. In other words, the molecules of arabic gum decrease the active center of dissolution of steel. Also, we can see in fig. 3 that the inhibitor impedance response reaches the maximum value of 83% efficiency for the concentration of 0.3%. We can therefore deduce that the 0.3% content has the best inhibition capacity. Nyquist impedance diagrams (Fig. 4) contain depressed semicircles with the centre under the real axis. Such a behaviour characteristic for solid electrodes and often called the frequency of the dispersion, could be attributed to different physical phenomena such as the roughness and inhomogeneities of solid surfaces, impurities, grain boundaries and the distribution of active sites on the surface [36]. Therefore, a constant phase element (CPE) instead of a capacitive element is used to obtain a more precise fit of the experimental data set. The impedance function of a CPE is defined by the following mathematical expression [13], [14], [37]:

$$Z_{CPE} = A^{-1} (j\omega)^{-n} \qquad (2)$$

where A is the constant CPE (in $\Omega^{-1} s^n / cm^2$), $\omega = 2\pi f$ is the angular frequency of sinusoidal modulation (in rad / s), $j^2 = -1$ is the imaginary number and (n) is an empirical exponent ($0 \leq n \leq 1$) which measures the deviation from the ideal capacitive behaviour [38], [39].

The CPE becomes the capacity when the phase shift coefficient is equal to 1. The double-layer capacity (Z_{dc}) has the expression (3):
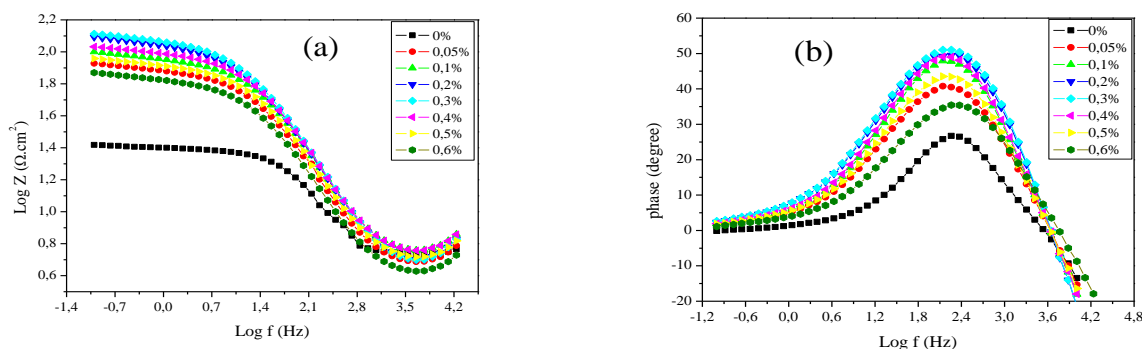
$$Z_{dl} = j/C\omega \qquad (3)$$

Fig. 4. Bode curves (a) and phase angle (b) of the corrosion of S235 steel
in 1M HCl at different concentrations of arabic gum.

Table (2) Impedance parameters and inhibition efficiency values for S235 steel
in 1 M HCl containing different concentrations of arabic gum.

C	R_s	R_{tc}	C_{dL}	N	τ	IE (%)	
(%)	($\Omega.cm^2$)	($\Omega.cm^2$)	($\mu F/cm^2$)		(s)	(SIE)	(Tafel)
0	5.83	21.51	119.11	0.77	0.00256	-	-
0.05	4.90	81.41	72.03	0.81	0.00586	73.59	72.79
0.1	5.79	95.22	61.93	0.83	0.00590	77.42	77.25
0.2	5.08	119.05	49.23	0.84	0,00586	82.00	81.86
0.3	4.67	128.52	46.81	0.88	0.00602	83.08	83.61
0.4	5.72	103.35	57.33	0.85	0.00592	79.19	79.44
0.5	5.21	86.62	68,51	0.82	0.00593	75.11	74.37
0.6	4.24	70.56	83,03	0.80	0.00586	69.53	67.O2

In the Table (2), the calculated time constant (τ) and the double-layer capacity (C_{dl}) derived from the CPE are also presented, using the following equations [40]:

$$C_{dl} = (A R_{ct}^{1-n})^{1/n} \qquad (4)$$

$$\tau = C_{dl} R_{ct} \qquad (5)$$

The impedance parameters, namely the resistance to charge transfer (R_{ct}), the constant phase element (A) linked to the capacity of the double-layer and the exponent (n), obtained for the capacitive semicircle S235 steel in 1 M HCl with or without arabic gum are shown in the Table (2).

These parameters were calculated from the non-linear least-squares adjustment of the equivalent circuit shown in fig. 5.
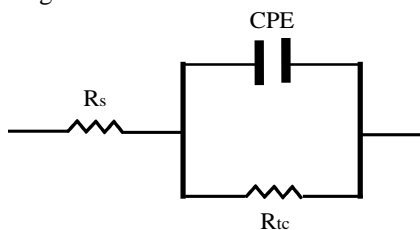

Fig. 5. Electrical circuit allowing the modeling
of the impedance spectra.

Analysis of the impedance results grouped in the table (2), shows that the value of resistance to charge transfer, R_{ct}, increases with the concentration of arabic gum and reaches a maximum value of 128.52 $\Omega.cm^2$ at 0.3% in mass concentration.

High resistance to charge transfer is associated with a slower corrosion system, due to a decrease in the active surface necessary for the corrosion reaction [41], [42]. The increase in the value of the empirical exponent (n) after the addition of arabic gum in the corrosive solution may corroborate this conclusion. Indeed, the lower value (n) for an uninhibited solution (n = 0.77) indicates a surface heterogeneity resulting from roughening and / or the formation of metal on the surface of the corrosion products. The values of (n) are between 0.80 and 0.88 in the case of inhibited solutions. The addition of arabic gum increases the value of (n) indicating a reduction in surface inhomogeneity due to the adsorption of molecules at the most active adsorption sites on the surface of S235 steel. Also, the addition of arabic gum to the corrosive solution decreases the capacity of the double-layer (C_{dl}). It increases the value of the time constant (τ) up to the 0.3% content (Table 2). Indeed, when the concentration is increased to 0.3% in the corrosive medium, the interface parameter (τ) increases from 0.00256 to 0.00602 s. At the same time the capacity value (C) decreases from 119.11 to 46.81 $\mu F / cm^2$,

which means that the charge and discharge rates at the metal-solution interface are considerably reduced. This shows that there is adequacy between the quantities of charge which can be stored; therefore the capacity and the rate of discharge in the interface (τ) [43]. The double-layer between the charged metal surface and the solution is considered to be an electrical capacitor. The adsorption of arabic gum on the surface of the metal decreases its electrical capacity because they displace water molecules and other ions initially adsorbed on the surface. The decrease in this capacity with the increase in gum concentrations can be attributed to the formation of a protective layer on the surface of the electrode [44]. The thickness of this protective layer increases with the increase in the concentration of inhibitor, also, the gum will adsorb electrostatically on the surface of the electrode, resulting in a noticeable reduction in C_{dl}. This trend conforms to the Helmholtz model, given by the following equation [45]:

$$C_{dl} = \varepsilon \varepsilon_0 / d \qquad (6)$$

where (d) is the thickness of the protective layer, ε is the dielectric constant of the protective layer and $\varepsilon 0$ is the permittivity of the free space (8.854×10^{-14} F / cm).

The inhibition efficiency, IE (%), is calculated from Rct. It is noted that IE (%) increases with the concentration of inhibitor (Table 2) to reach a maximum value of 83% for a concentration of 0.3%. However, beyond this content, the provision of arabic gum inhibitors decreases.

3.2.2. Adsorption isotherm and surface analysis

It is assumed that the action of an inhibitor in aggressive acid media is due to its adsorption at the metal / solution interface. The adsorption process depends on the electronic characteristics of the inhibitor, the nature of the metal surface, temperature, steric effects and varying degrees of surface-site activity [46]. The establishment of isotherms that describe the adsorption behaviour of corrosion inhibitors is essential because they provide important clues to the nature of the metal-inhibitor interaction. The values of the degree of surface coverage (θ) corresponding to the different concentrations of gum arabic were used to determine which isotherm best describes the adsorption processes. In the present study, the values of (θ) were calculated using the impedance results according to the following equation [47]:

$$\theta = (R_{ct} - R_{ct}^0) / R_P \qquad (7)$$

where R_{ct}^0 and R_{ct} are the values of resistance to charge transfer without and with inhibitor, respectively. The results obtained for arabic gum in a 1

M HCl solution correspond well to the Langmuir adsorption isotherm given by (8) [48]:

$$C_{inh}/\theta = 1/K_{ads} + C_{inh} \qquad (8)$$

where θ is the degree of surface coverage and C_{inh} is the concentration of inhibitor in the electrolyte. K_{ads} is the equilibrium constant of the adsorption process and is related to the standard Gibbs adsorption energy, ΔG_{ads}^0, according to [49]:

$$\Delta G_{ads}^0 = -RT\ln (10^6 K_{ads}) \qquad (9)$$

where 1.10^6 is the concentration in molecules of water expressed in (mg / L), R (J./K/mol) the constant of perfect gas and T (K) the absolute temperature.
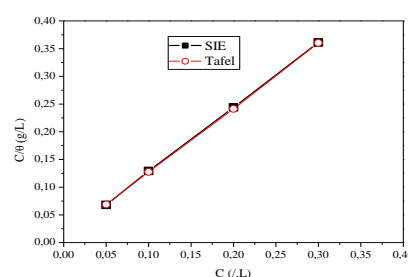

Fig. 6. Langmuir adsorption traces for S235 steel in 1M HCl containing arabic gum.

Fig. 6 shows the dependence of C_{inh} / θ on C_{inh}. To calculate the adsorption parameters, straight lines were drawn using the method of least squares. The isotherms are shown in fig. 6.

The results are presented in table (3). A perfect fit is observed for concentrations between 0.05% and 0.3%, with the regression coefficients up to 0.9999 and the lines obtained have slopes very close to unity, which suggests that the experimental data are well described by the Langmuir isotherm and have a monolayer adsorption characteristic. This type of isotherm implies the hypothesis of no interaction between the species adsorbed on the surface of the electrode [50]. The K_{ads} values were calculated (8) and given in table (3). The K_{ads} values can be considered as a measure of the adsorption forces between the inhibitory molecules and the metallic surface [51]. Consequently, the most potent interaction between the double-layer existing at the phase boundary and the adsorbent molecules and the highest IE (%) is due to arabic gum (Table 3).

The calculated values of ΔG_{ads}^0, using (9), are also given in Table 3. The negative values of ΔG_{ads}^0 ensure the spontaneity of the adsorption process and the stability of the layer adsorbed on the surface of steel.

In general, the type of adsorption is considered to be a physisorption if the absolute value of ΔG_{ads}^0 is of the order of 20 kJ / mol or less. The inhibitory behaviour is

attributed to the electrostatic interaction between organic molecules and the iron atom. When the absolute value of ΔG_{ads}^0 is of the order of 40 kJ / mol or more, the adsorption can be considered as a chemisorption. In this process, the covalent bond is formed by the sharing or the transfer of charge of the inhibitory molecules to the metallic surface [52], [53]. Based on the literature [54], the values of ΔG_{ads}^0 calculated in this work (table 3) indicate that the adsorption mechanism of arabic gum on metallic structural steel S235 in a solution of HCl 1 M is typical of physisorption.

The same conclusion was given regarding the interaction of mild steel with the mercapto-triazole compound and 3, 4-dihydropyrimidin-2 (1H) -ones, respectively by Wang et al. [55] and Yadav et al. [56]. The large negative value of ΔG_{ads}^0 in arabic gum indicates that this inhibitor is strongly adsorbed on the surface of steel [57], [58].

Table (3) The different thermodynamic values of the Langmuir isotherm of arabic gum for S235 steel in 1M HCl.

4. CONCLUSION

During this work, we studied the corrosion inhibition behavior of S235 steel in a hydrochloric acid (HCl) solution of 1M concentration in order to protect installations against corrosion. This study was carried out electrochemically.

Corrodability tests were performed using Tafel polarization and by electrochemical impedance spectroscopy measurements. The study showed that the rate of corrosion is reduced by about 83% through the use of a natural inhibitor, arabic gum. The results obtained show that this inhibitor acts as a mixed inhibitor and has an efficiency of around 83% for an optimal concentration of 0.3%. The evolution of the impedance diagrams in the Nyquist plane as a function of the concentrations of natural inhibitor confirms the formation of a protective layer, which results in the increase in the charge transfer resistance together with a decrease in the capacity of double layer. The results obtained by the Tafel method are in agreement with those obtained by electrochemical impedance spectroscopy measurements with an inhibitory efficiency of the order of 83% to 0.3% of arabic gum.

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R^2		Slope		K$_{ads}$ (L/g)		ΔG_{ads}^0(kJ/mol)	
(SIE)	(Tafel)	(SIE)	(Tafel)	(SIE)	(Tafel)	(SIE)	(Tafel)
0.999	0.999	1.17	1.16	9.85	10.54	-22.77	-22.93

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